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MASS SPECTRAL ANALYSIS OF ROCKET ENGINE COMBUSTION PRODUCTS CONTAINING BORON NITRIDE

TECHNICAL DOCUMENTARY REPORT NO. SSD-TDR-62-202

DECEMBER 1962

ROCKET RESEARCH LABORATORIES
EDWARDS, CALIFORNIA
AIR FORCE SPACE SYSTEM DIVISION
AIRFORCE SYSTEM COMMAND
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NOTICES

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This report has been reviewed and approved

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a = constant, independent of magnetic field.

Mi = mass at distance Xm from Xo.

The limit of error for the mass determinations is ±0.003 mass units. Nearly all of the identified mass fragments were within this limit.

Identified mass fragments in the sample are presented in Appendix I. The actual mass fragments and computed mass fragments are given. Reference lines (Ma, b) used for mass fragment calculations were:

$^{16}O^{+2}$	7.9974
$^{65}Cu^{+4}$	16.2319
$^{23}Na^{+}$	22.9898
$^{28}N_2^{+}$	28.0061
$^{64}Cu^{+2}$	32.4639
$^{37}Cl^{+}$	36.9659

The ion intensities of the major mass fragments of interest were accomplished by determing the percent transmittancies of the major mass lines on the plate using a Jarrell Ash Densitometer. The percent transmittancies were converted to energy values by using equation 2[3].

$$\text{Log } E_T = \frac{\text{Log } \dfrac{1 - Tm}{Tm - Ts}}{R} \qquad (2)$$

Where E_T = energy at % Tm.

Tm = transmittancy of line mass "M".

Ts = maximum line density (minimum T) for plate.

R = plate emulsion constant obtained from standard titanium transmittancy vs. exposure curves.

The constants R and Ts were determined experimentally from multiple exposures of spectrographic grade titanium electrodes on the same plate.

Relative transmittancies of the major mass lines of interest converted to ion intensity values are given in Appendix II.

Corrected ion intensity values of the mass fragments in Appendix II were calculated using sensitivities for ^{10}B, ^{11}B, ^{14}N and ^{16}O obtained from experimentally derived plate sensitivities of pure boron, boron nitride and boron oxide. All sensitivities were calculated with reference to the relative sensitivity of $^{11}B = 1.0$. The corrected ion intensities were calculated using Equation 3.

$$Ec = Em \times Sm \times Im \qquad (3)$$

Where Ec = corrected ion intensity of mass fragment "M".

Em = relative ion intensity value for "M" obtained from plate calibration curve.

Sm = sensitivity of mass fragment relative to $^{11}B = 1.0$

$Sm = K^{11}B/Km$

Im = Isotopic abundance of mass "M".

Values for mass fragment sensitivities were found to be:

$K^{10}B$	=	.33	$K^{14}N$	=	.55
$K^{11}B$	=	1.00	$K^{16}O$	=	.40

The reproducibility of these sensitivity constants is ±5.0 percent of the mean value.

Corrected ion intensity values are presented in Appendix III. Hydrocarbon, zirconium, and other fragment ion intensities were not determined.

All mass fragment intensities increased linearly as the exposure increased. Mass fragment intensities due to adsorbed gases decrease with exposure because sample heating drives these gases off during initial sparking. This indicates that the fragments found were associated or bonded as in a solid adduct or polymeric -BNH- type compound.

CONCLUSION

Analysis by Kjeldahl method [1] showed 49 percent by weight nitrogen for the engine residue under consideration in this paper. Therefore, the total boron nitride would be 89 percent by weight in the residue. However, a part of the nitrogen is bonded to hydrogen and in the -BNH- polymeric form. The ion intensities in Appendix III may be correlated in the following manner:

1. Nitrogen is present to the extent of 6.14 relative intensity units in an uncombined fragment state in the spark.

2. Boron (sum of ^{10}B, ^{11}B, ^{11}BH, ^{11}BO) equals 9.12 units from Appendix III.

Since boron nitride will not exhibit a strong BN^+ fragment (bond energy considerations decrease the probability of this fragment in the high energy spark), all of the free nitrogen fragments were assumed to come from boron nitride.

The following breakdown of ion intensity units is given:

Fragment Contribution	B	N	Total Units
BN	6.14	6.14	12.28
NH, BNH, BH	1.91	1.91	3.82
BO	.32	--	.32
Residual B	.75	--	.75

The sum of this unit counting method is 17.17 relative intensity units (excluding oxygen) so that a percentage composition may be postulated thus:

BN	=	71.6% (atomic)
-BNH-	=	22.2%
BO	=	1.8%
B	=	4.4%

where oxygen, hydrocarbons, zirconium, etc. ion intensity contributions are excluded from the calculations. This analysis accounts for the distribution of boron between boron nitride and polymeric boron-nitrogen-hydrogen compounds which would otherwise not have been accomplished by Kjeldahl method.

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2. Consolidated Electrodynamics Corp. Mass Spectrometer 21-110 Manual, 1961.

3. Hull, Charles W., "Photographic Quantitative Anaylsis with a Solids Spark Mass Spectrograph," E-14 Mass Spectrographic Conference, May 1962.

APPENDIX I

The computed mass vs. true mass of identified mass fragments.

Mass (Computed)	Mass (True, ^{12}C = 12.0000)	Mass (Fragment)
10.0163	10.0129	^{10}B
11.0160	11.0093	^{11}B
12.0011	12.0000	^{12}C
12.0173	12.0171	^{11}BH
13.0046	13.0078	^{12}CH
13.0244	13.0249	$^{11}BH_2$
14.0110	14.0141	$^{12}CH_2$
14.0001	14.0031	^{14}N
15.0015	15.0001	^{15}N
15.0143	15.0109	^{14}NH
15.9991	15.9950	^{16}O
16.0173	16.0187	$^{14}NH_2$
17.0057	17.0027	OH
17.0283	17.0265	$^{14}NH_3$
18.0138	18.0106	HOH
18.0354	18.0313	$^{14}NH_4$
19.0037	18.9984	^{19}F
22.9896	22.9898	^{23}Na
23.9859	23.9850	^{24}Mg
25.0101	25.0123	$^{11}B\,^{14}N$
26.0104	26.0078	^{10}BO
26.9861	26.9815	^{27}Al
27.0089	27.0042	^{11}BO
27.9770	27.9769	^{28}Si
28.0060	28.0062	$^{14}N_2$
29.0123	29.0139	$^{14}N_2H$

APPENDIX I (Cont'd)

Mass (Computed)	Mass (True, $^{12}C = 12.0000$)	Mass (Fragment)
31.9752	31.9721	^{32}S
34.9669	34.9688	^{35}Cl
35.0196	35.0253	$^{10}B-^{11}B-^{14}N$
36.0188	36.0217	$^{11}B_2-^{14}N$
36.9657	36.9659	^{37}Cl
39.9696	39.9626	^{40}Ca
89.9093	89.9043	^{90}Zr
44.9774	44.9817	$^{90}Zr^{+2}$
29.9689	29.9680	$^{90}Zr^{+3}$

Reference lines used for mass fragment calculations were:

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APPENDIX II

Ion intensities from major mass fragment line relative transmittancies.

M	E_M
^{10}B	2.39
^{11}B	9.00
^{11}BH	.16
^{14}N	3.38
^{14}NH	.23
$^{14}NH_2$	.19
$^{14}NH_3$	.39
$^{14}NH_4$	.24
^{10}BO	.21
^{11}BO	.25
^{16}O	.88

APPENDIX III

Corrected Mass Fragment Ion Intnesity Values

M	Ec	M	Ec
^{10}B	1.38	$^{14}NH_2$	.34
^{11}B	7.29	$^{14}NH_3$	.71
^{11}BH	.13	$^{14}NH_4$	.44
^{14}N	6.14	^{10}BO	.12
^{14}NH	.42	^{11}BO	.20
		^{16}O	2.20

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Unclassified report

Rocket engine combustion products containing boron nitride were analyzed by spark ion source vacuum mass spectrometry. Molecular species found in the residue are identified and reported in tables of actual atomic weight vs. the computed atomic weight. The quantitative analysis of the residue and a discussion of the method of quantitative calculation is presented.

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